A high speed interferometric dilatometer based on inductive heating of the specimen

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Abstract. A high speed dilatometer has been designed for high precision measurements of the thermal expansion/contraction of solid samples. The length of the specimens is measured by an differential interference method with a resolution of 0.3 nm and a reproducibility of 30 nm. The temperature is controlled by induction heating and gas cooling. The maximum heating rate is about 100 K/s, the maximum cooling rate 50 K/s. Electrically non-conductive specimens can be heated indirectly via metallic rings around the specimen. The system enables measurements under different gas atmospheres, including oxygen up to temperatures of 1600°C.

Keywords: optical dilatometer, induction heating, laser interferometer

1. Introduction

Dilatometers are generally used for measuring the length of a specimen within a temperature range. This allows to derive the coefficient of thermal expansion (CTE) and to find material phase changes. The aim of this study is to design and test a high speed high precision optical dilatometer based on inductive heating of electrically conductive solid specimens for temperatures up to 1600°C.

A typical dilatometer heats the specimen using a furnace. The mass of the furnace is considerably larger than the mass of the specimen. That limits the heating speed to about 1 K/s. After having taken a measurement, much time is needed for cooling down the furnace, before the following measurement can be started. Unfortunately the heat the furnace produces also heats the dilatometer as a whole. Although this is a slow process but it causes drifting, which is to be a remarkable disadvantage when a series of measurements is to be taken. That makes it desirable to focus the heat on the specimen in order to achieve a higher heating rate and a better accuracy and reproducibility as well.

For measuring of the length of the specimen mechanical and optical principles are known. James *et al* [1] gave a survey of measurement techniques of dilatometers. Widely used are silica push rods that touch the specimen and measure its elongation using a linear variable differential transformer (LVDT) displacement transducer. The thermal expansions of the push rod and of the specimen's support have some influence on the readings. Therefore the behavior of the support and the push rod must be known and it must be reproducible. Push rod dilatometers enable a resolution of 10 nm and a reproducibility of 150 nm to be achieved.

For high precision measurements optical measurement techniques are considered appropriate, based on the optical interference of light reflected from the specimen. Laser interferometric dilatometers were proposed in the 1970s [2]. The lack of adequate mirrors limited the temperature to 1350 K maximum. Furnaces were used for heating. From 2000, Okaij *et al* [3] and Watanabe *et al* [4] have introduced a series of laser interferometric dilatometers to measure the CTE of solids. In 2005 Tseng and Jiang [7] presented an optical interference dilatometer based on shadow moiré technique. [8] *et al* designed an optical dilatometer based on a Michelson interferometer for the 4...300 K temperature range.

The well-established optical arrangements require an optical path of constant or at least stationary refractive index with the same distribution at the way to and from the specimen. That is ensured by using a furnace for heating the gas around the sample, or by placing the sample in a vacuum. Unfortunately an inductively heated sample heats the surrounding gas by conduction so that convection will emerge. Steep gradients of temperature will occur, and of the refractive index as well as the refractive index of a gas depends on its density. Hence the gas around the specimen distorts the measurement. Calibration is not helpful due to the dependence of the temperature field on the gas mixture and on the heating and cooling rates. Therefore, for fast inductive heating of

the sample together with optical measurement, an arrangement is needed that eliminates the influence of the gas atmosphere around the specimen.

2. Device description

Figure 1 shows a schematic of the interferometric dilatometer with inductive heating of the sample. Basically, it includes the following modules: the gas chamber with the specimen holder, the inductive heating device with a HF power generator, a coil and so-called robber rings, the gas shower for cooling the sample, the double beam differential laser interferometer with two light guide push rods, and the control and data acquisition unit. The modules are described in detail in the following.

2.1. Inductive heating

The inductive heating assembly consists of a high frequency (HF) power generator, a fixed heating coil and two height-movable robber rings to form the electro-magnetic field. Both the heating coil and the robber rings are water-cooled. The specimen to be measured is located on the axis of the heating coil and robber rings whereas the silica tube is slightly shifted out of the axis to contain both light guide rods.

The working frequency of the induction heating coil is about 200 kHz. Hence the depth of penetration δ of the eddy currents is not greater than about 100 μm [9]:

$$\delta = \frac{1}{2\pi}\sqrt{\frac{\rho \cdot 10^7}{f \cdot \mu}} \qquad \text{in mm,} \tag{1}$$

with the electrical resistivity ρ and the magnetic permeability μ of the material and the generator frequency f. Therefore heat is induced close to the surface of the specimen. The corpus of the specimen is mainly heated by diffusion. At the edges of cylindrical specimens the temperature will be raised. This is shown by a FEA simulation in Figure 2. Local field weakening by the robber rings counteracts the temperature raising and reduces the temperature difference along the specimen. Figure 3 and 4 illustrate this by the example of a specimen made of 1.4301 steel. The optimum position of the robber rings for a minimum of the temperature difference along the surface of the specimen depends on the dimensions and on the material of the specimen as shown in Figure 5. In optimum position of the robber rings, the temperature at the edges of the specimen is equal to that in the middle of the cylinder surface. However, a disadvantage of the robber rings is that they reduce the maximum heating rate and get heated up themselves.

The temperature difference ΔT emerging between the inner axis of the specimen and its circumference depends on the specific heat capacity c_p, the density ρ, the thermal conductivity λ and the radius R of the sample. Deduced from the heat equation in cylindrical coordinates, the radial temperature difference ΔT_r is [10]:

$$\Delta T = \frac{\rho c_p \mathrm{d}T/\mathrm{d}t}{4\lambda} R^2 \tag{2}$$

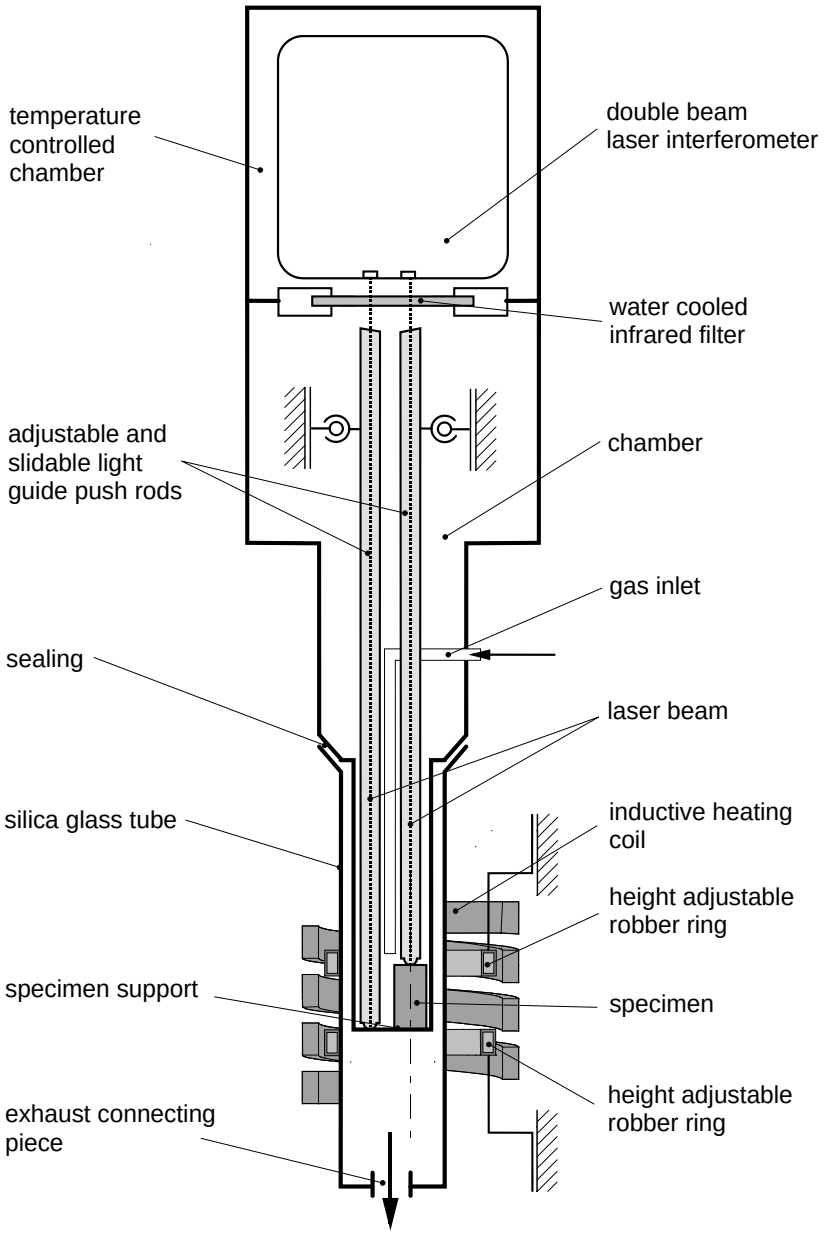


Figure 1. Schematic of the interferometric dilatometer with inductive heating of the sample

for a given temperature rate $\mathrm{d}T/\mathrm{d}t$ at the circumference of the sample. This relation reasonable limits the heat rates for measurements.

2.2. Gas cooling

The cooling gas is applied to the sample by a gas shower. The gas is fed through a tube out of fused silica to a couple of nozzles towards the sample to be cooled. The amount of cooling gas is controlled by a proportional valve, according the cooling requirements

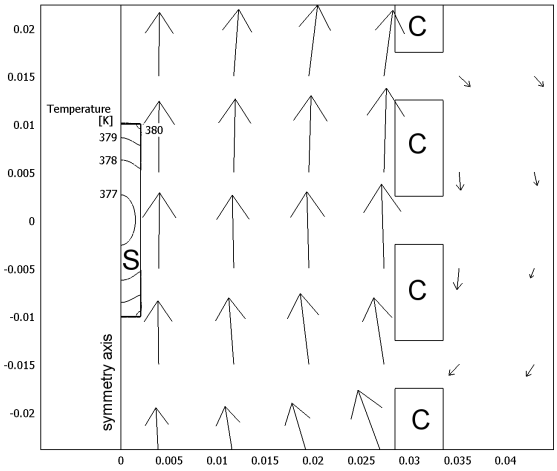


Figure 2. Temperature field of an inductively heated cylindrical (R2 x 20) 1.4301 steel sample, computed using a FEA model; S – specimen, C – windings of the heating coil

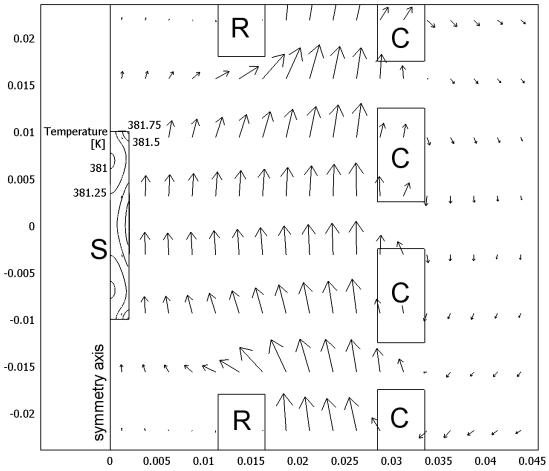


Figure 3. Temperature field of the specimen of Fig. 2 with temperature differences reduced by robber rings, computed with a FEA model; S – specimen, C – windings of the heating coil, R – robber rings

of the temperature profile. Usually Helium is used for high cooling capacity, due to it's high heat conductivity and heat capacity. To work at sub ambient temperatures, cold nitrogen, drawn from a dewar with liquid nitrogen can also be used. Figure 6.a shows the gas shower inside of the protection tube without the sample-holder. In Figure 6.b, the sample holder is in it's working position and the light guide push rods are put on the heated sample and on the sample holder respectively.

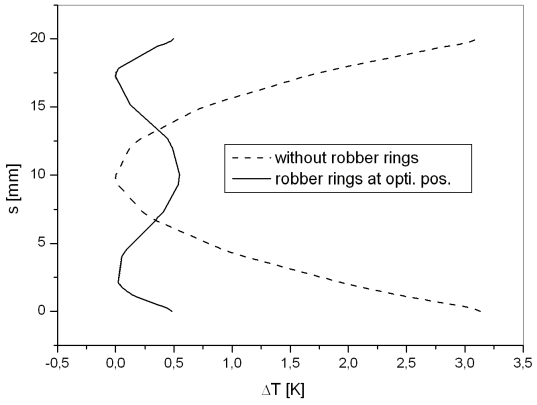


Figure 4. Temperature difference ΔT along the outer side of a cylindrical specimen s with and without robber rings, arrangement according to Fig. 2 and resp. Fig. 3

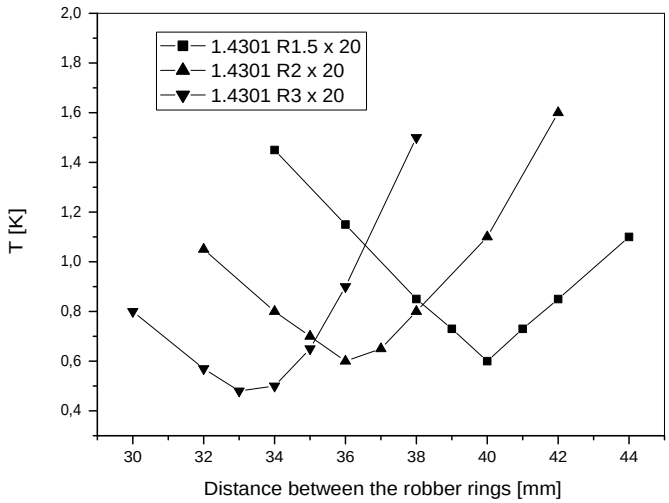
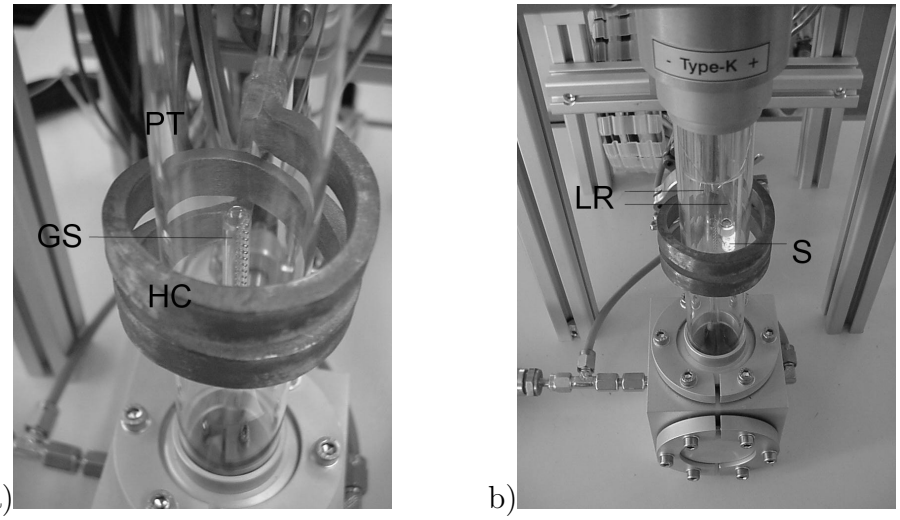


Figure 5. Temperature difference along the outer side of a cylindrical specimen ΔT depending of the positions of the robber rings P

2.3. Optomechanics

As described before the interferometric measurement is complicated by steep and unknown temperature gradients around the sample. For the same reason, a conventional push rod measurement would be inaccurate. Therefore, we designed the optomechanical assembly such that the laser beams solely pass through evacuated parts on the whole optical path. This is reached by two light guide push rods touching both the specimen and the support of the specimen for a differential measuring. The laser beam is reflected at the bottom inner surface of the push rods. Thus, heat transfer from the specimen tho the push rods and to the gas around the specimen cannot cause a change of the



a) b)

Figure 6. Sample holder and gas shower of the dilatometer, a) sample holder removed, PT – protection tube, HC – heating coil, GS – gas shower, b) sample holder in working position, S – sample, LR – light guide push rods

refractive index along the optical path. Solely the elongation of the push rods due to their heating will only insignificantly change the gas/vacuum length ratio of the optical path.

The push rods are angle-adjustable and height-slidable. Between the chamber where specimen support, specimen, and light guide push rods are arranged and the temperature controlled chamber of the laser interferometer, a water cooled infrared filter is placed.

2.4. Control and data acquisition

A block diagram of the control unit for temperature control and data aquisition is shown in Figure 7. The control of the power output of the RF-Generator and the amount of cooling gas is done by a PID controller. To optimize the behavior of the closed loop control, a forward calculation of the output signal is made by a mathematical model, according the heating or cooling rate and the actual set temperature. To control the proportional valve for gas cooling, the output signal of the controller is inverted, and a bias is added for cooling, to get some overlap between heating and cooling, because of the faster response of the RF-generator as the gas cooling. The cycle time of the controller is 1ms (1kHz), the same as the maximum acquisition rate of the temperature signal. A 16 Bit controller is used for data acquisition, temperature control and communication with the host computer.

3. Test of the device

We tested a specimen of stainless steel (1.4301) shaped as a hollow cylinder (inner radius 4 mm, outer radius 6 mm, length 10 mm). The exact denotation is ATI W.1;4301 6x1 DIN 17457 PK1 K2 melt 653330 DOCQ. Figure 8 shows the nominal and the measured

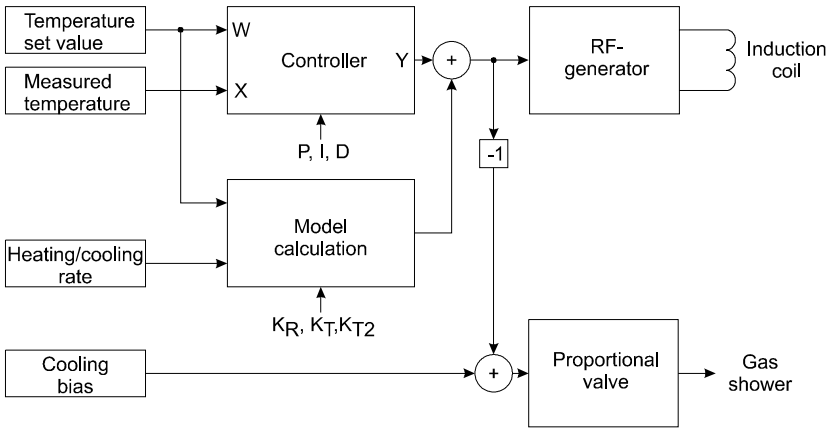


Figure 7. Block diagram of the control unit of the dilatometer

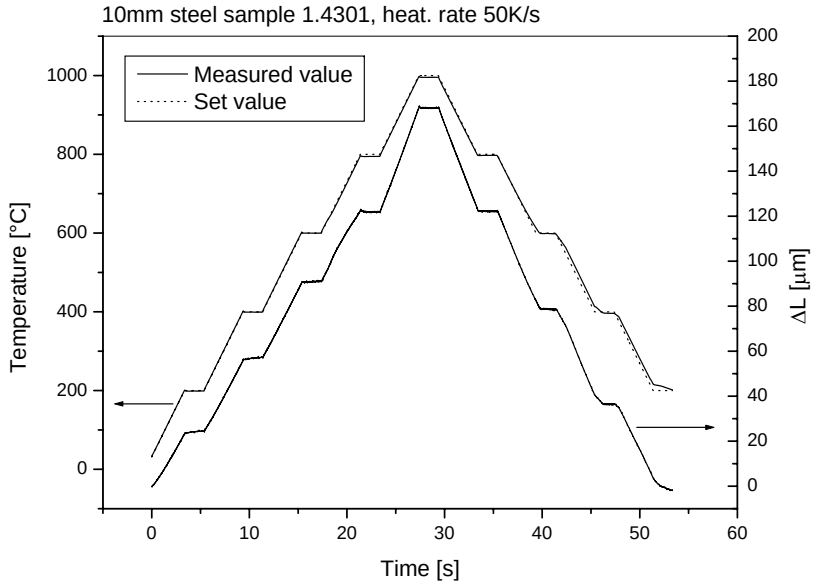


Figure 8. Nominal and measured temperature run of a specimen of stainless steel (1.4301) in form of a hollow cylinder (inner radius 4 mm, outer radius 6 mm, length 10 mm)

temperature runs, detected by a K-type thermocouple bonded to the sample. The heating rate and the cooling rate are 50 K/s in steps of 200 K separated by dwell times of 2 s. Since the heat flux is not sufficient to reach the nominal cooling rate when the temperature of the sample is near of that of the cooling gas, the measured temperature run cannot follow the nominal temperature in the end.

Figure 9 shows the elongation over the temperature of a 20 mm fused silica rod (NIST SRM 739) with a heating-rate of 5 K/min. Data from literature are pasted in.

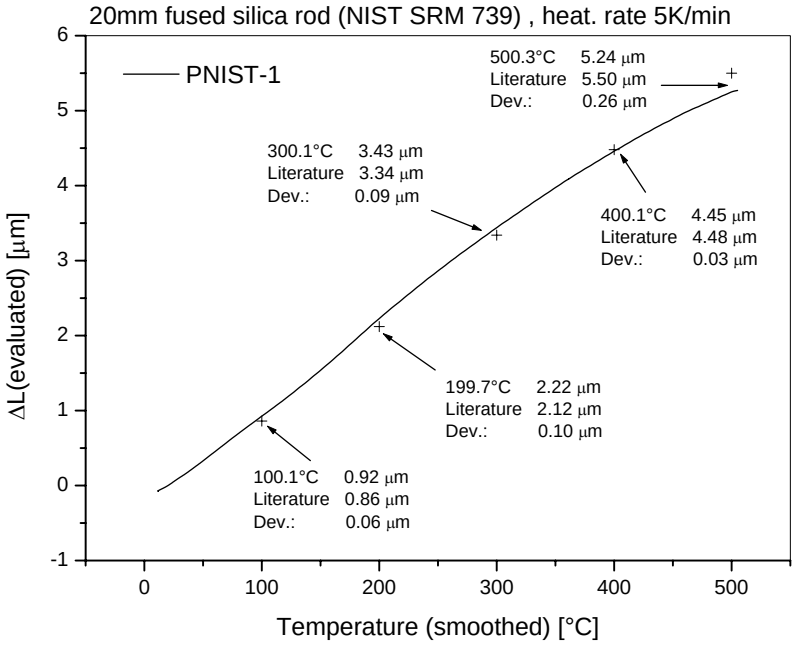


Figure 9. Measurement of a 20 mm fused silica rod, heating-rate 5 K/min

4. Conclusion

We have described a dilatometer that can measure displacements by an differential interference method with a resolution of 0.3 nm. The temperature of the specimen is controlled by induction heating and gas cooling. Hence heating rates up to 100 K/s and cooling rates up to 50 K/s can be performed. The apparatus is especially suitable for the study of electrically conductive solid materials under different gas atmospheres, including oxygen up to temperatures of 1600°C.

The interferometric length measurement can also be combined with a conventional furnace. Such arrangement would achieve lower heating rates. It is suitable for the measurement of electrically non-conductive samples of materials with a small coefficient of thermal expansion (CTE).

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